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Effects of heat treatment on microstructure and mechanical properties of Mg−5Zn−0.63Er alloy

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Abstract: The microstructure evolution of the Mg−5Zn−0.63Er (mass fraction, %) alloy containing quasicrystalline phase (I-phase) under the as-cast condition was investigated via different heat treatments. The results show that apart from the precipitation of the W-phase particles, the I-phase almost dissolves into the matrix after solid solution treatment at 480 °C for 10 h (T4 state). The solution-treated alloy was aged at 175 °C for 6−100 h (T6 state). The ultimate tensile strength of the peak-aged alloy is approximately 261 MPa companying with an elongation of 10.5%. The improvement of the tensile strength is mainly attributed to the presence of the rod-like $MgZn_2$ particles.
Key words: Mg−5Zn−0.63Er alloy; rare earth; Er; heat treatment; microstructure; mechanical properties

1 Introduction

Mg-based alloys containing rare earth metals are becoming more and more attractive due to their high strength and creep resistance [1−4]. One important series of the magnesium alloys are the alloys containing rare earth (RE) elements, such as Mg−Al−RE, Mg−Y−RE−Zr and Mg−Zn−RE alloys [5−7]. The magnesium alloys with RE addition are usually developed as heat-resisting alloys, which can widen the range of the applied temperature. It was reported that a thermally stable icosahedral quasicrystalline phase (I-phase) in the Mg−Zn−Y alloys, with a chemical composition of Mg3Zn6Y1, formed in-situ as a secondary phase during the solidification [8−14]. Also, a precipitated I-phase with a spherical morphology distributing at the matrix is observed during the heat treatment. The spherical phase is so stable that it can enhance the mechanical properties of the alloys, especially for the mechanical properties at elevated temperature via inhibiting the grain growth effectively [15].

The Er element has a high solubility in magnesium. According to the Mg−Er phase diagram, the solubility of Er in magnesium is as high as 6.9% (mole fraction) at a eutectic temperature of 570 °C, while it decreases to 3.17% (mole fraction) as the temperature drops to 300 °C. The change of the solubility has a great effect on the improvement of the mechanical properties through solution strengthening and precipitation hardening. Recently, the microstructure of the as-cast Mg−Zn−Er alloy has been investigated and the I-phase has been found [16]. However, until now, little work focused on the effects of heat treatment on the microstructure evolution and the mechanical properties of Mg−Zn−Er alloys. Therefore, in order to investigate the influence of the solid solution temperature on the microstructure and aging behaviors, the cast Mg−5Zn−0.63Er alloys were solid solution-treated at 440, 460, 480, 500 °C for 10 h, respectively, and then aged at 175 °C for 6−100 h.

2 Experimental

Alloys with a nominal composition of Mg−5Zn−0.63Er (mass fraction, %) were prepared from Mg (purity≥99.9%), Zn (purity≥99.9%) and Mg−30%Er master alloy in a graphite crucible under anti-oxidizing flux in an electric resistance furnace. The melt was poured into a steel mould and then cooled down in air. Ingots were solution heat-treated at 440, 460, 480 and 500 °C for 10 h and then quenched into cold water. After the solution treatment, the specimens were isothermally aged at 175 °C. The phase constituents were identified by X-ray diffraction (XRD) with Cu K_α radiation. The

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microstructures were analyzed by an optical microscope (OM) and a scanning electron microscope (SEM) equipped with an energy dispersive spectroscope (EDS). TEM specimens were prepared by electro-polishing and ion beam milling at an incident angle less than 10°, and electro-polishing of the samples was operated in a solution of 20% (volume fraction) nitric acid and 80% (volume fraction) methanol, using liquid nitrogen cold stage to prevent heating. The aging response of the specimens was observed by the hardness measurement. Vickers hardness was tested by an HXD−1000 Vickers hardness tester at a load of 0.98 N and a dwell time of 10 s, and more than eight measurements were collected for one sample.

3 Results and discussion

3.1 Microstructures of as-cast Mg−5Zn−0.63Er alloy

The phase compositions of the Mg−5Zn−0.63Er were identified by XRD. The XRD pattern of the alloy is shown in Fig. 1. It is suggested that the phase constituents are mainly composed of α-Mg solid solution, I-phase and Mg_4Zn_7 phase. As reported previously, the I-phase usually precipitates via the eutectic reaction when the addition of Y is more than 4% (mole fraction) in the Zn-rich alloys [17,18]. However, the I-phase is so brittle that it cannot be used as structural materials directly. YI et al [19] found that the I-phase coexists with the matrix as a eutectic in the as-cast Mg−Zn−Y alloys. Subsequently, the I-phase was observed in the Mg−Zn−Y, Mg−Zn−Gd and Mg−Zn−Er series alloys [20,21].

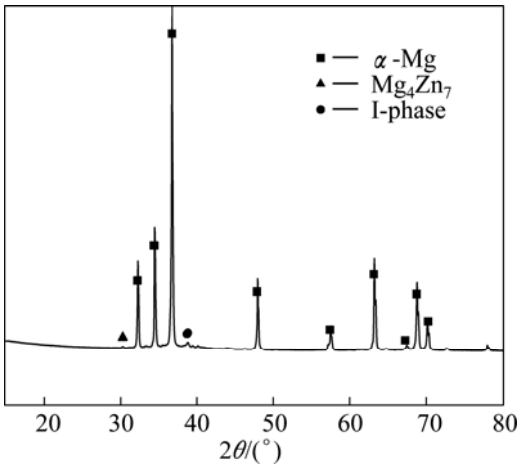

Fig. 1 XRD pattern of as-cast Mg−5Zn−0.63Er alloy

Figure 2 shows the SEM images of the Mg−5Zn−0.63Er alloy. The cast alloy contains dendrites of the α-Mg matrix companying with lots of compounds, as shown in Fig. 2(a). The micrograph at a higher magnification is obtained, as shown in Fig. 2(b). Three kinds of the compounds identified from the morphology can be found, which are marked by *B*, *C* and *D*. The EDS results of points *B*, *C* and *D* together with the matrix marked by *A* are listed in Table 1. It is indicated that the chemical composition of point *B* is close to the Mg_4Zn_7 phase. However, the chemical compositions of points *C* and *D* are near to the I-phase. This further confirms the existence of the I-phase which is mainly located at the grain boundaries.

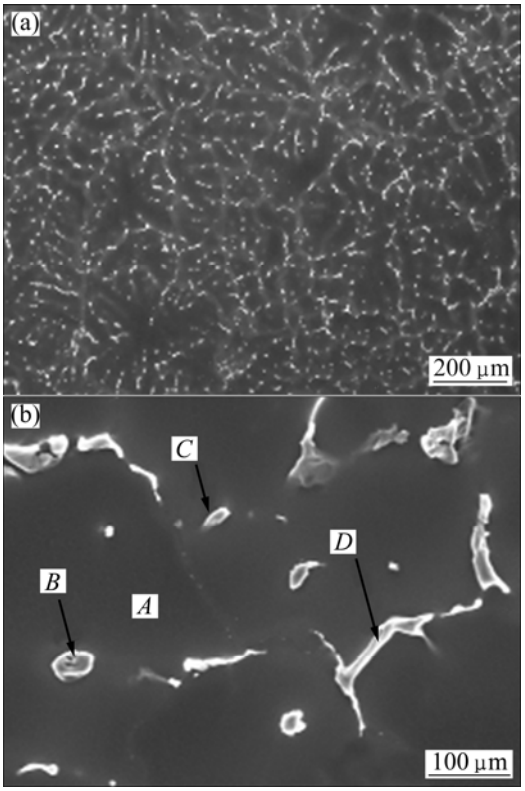

Fig. 2 SEM images of as-cast Mg−5Zn−0.63Er alloy: (a) Low magnification; (b) High magnification

Table 1 EDS results of as-cast Mg−5Zn−0.63Er alloy in Fig. 2(b)

Position	Composition (mole fraction)/%		
	Mg	Zn	Er
Matrix *A*	97.55	2.45	0
Point *B*	31.51	68.49	0
Point *C*	86.31	11.31	2.38
Point *D*	87.96	10.08	1.96

Figure 3 displays the TEM images of the I-phase and the corresponding SAED patterns observed in the as-cast Mg−5Zn−0.63Er alloy. It is well known that the diffraction pattern showing 5-fold symmetry is typical for the icosahedral quasicrystalline structure [10,22], and the I-phase ($Mg_{30}Zn_{60}Er_{10}$) is found in Mg−Zn−Er alloy [16]. Figure 3(b) shows the SAED pattern of the I-phase with its 5-fold symmetry. Besides, the SAED patterns of the I-phase with 3-fold symmetry and 2-fold symmetry are observed, as seen in Figs. 3(c) and (d). The EDS

analysis indicates that the composition of point *A* is approximately 30.05%Mg−58.97%Zn−9.98%Er (mole fraction), which is corresponding to the stoichiometric composition of the $Mg_{30}Zn_{60}Er_{10}$ phase.

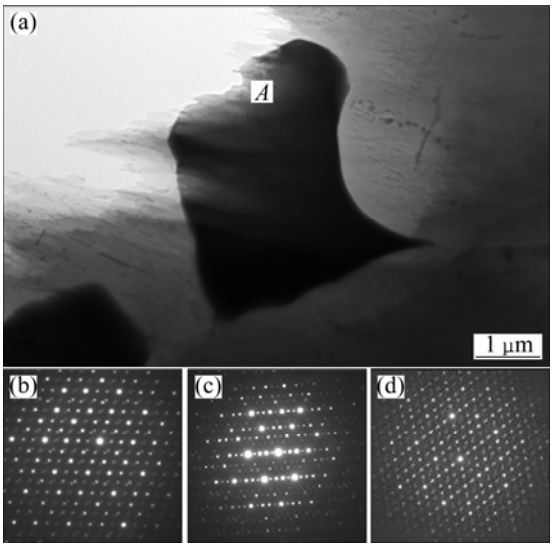

Fig. 3 TEM images of as-cast Mg−5Zn−0.63Er alloy: (a) Bright field image; (b), (c), (d) Corresponding SAED patterns for position *A* in (a), which represent 5-fold symmetry (b), 3-fold symmetry (c) and 2-fold symmetry (d)

3.2 Microstructures of solution-treated alloys

Figure 4 shows the XRD patterns of the solid solution-treated alloys. As shown in the XRD patterns, it is found obviously that the solid solution treatment at 440 °C has no great influence on the dissolution of the I-phase into the matrix. The peaks corresponding to the I-phase can be seen in the XRD pattern. It should be noted that the peaks corresponding to the W-phase appear. It is implied that the W-phase is apt to precipitate from the matrix during the solid solution treatment. Previous research reported that I-phase (icosahedral quasi-crystalline structure) and W-phase (face-centered cubic) are the main ternary equilibrium phases in the as-cast Mg−Zn−Y alloys [23,24]. During the solid solution treatment, element Er aggregates gradually in the matrix, whereas the area containing higher Er content can meet the requirement of precipitation of the W-phase. As the temperature increases, the strength of the peaks corresponding to the I-phase becomes weak. Remarkably, the peaks corresponding to the I-phase cannot be found within the sensitivity limits of X-ray diffraction after solid solution treatment at 480 and 500 °C, which suggests that the I-phase may decompose and dissolve into the matrix mostly.

Figure 5 shows the OM images of the solid solution-treated alloys. The content of the secondary phase decreases and the average grain size is (200±20) μm after solid solution treatment at 440 °C for 10 h. However, the I-phase does not dissolve into the *α*-Mg matrix completely because of the low solution temperature. As the temperature further increases up to 460 °C, more secondary phases dissolve into the *α*-Mg matrix and the average grain size is (300±20) μm. At 480 °C, the secondary phases distributing at the grain boundaries almost disappear. The grain boundaries are clear relatively, and the average grain size is (300±20) μm. Obviously, the high solid solution temperature of 500 °C has a great effect on speeding the grain growth of the alloy, and the average grain size is (500±20) μm. On the whole, the heat treatment technology plays an important role in dissolving the coarse secondary phases into the matrix and making the precipitation of the tiny W-phase apart from speeding the growth of the grains.

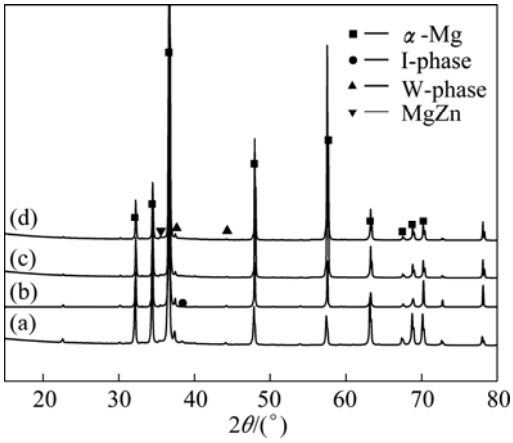

Fig. 4 XRD patterns of Mg−5Zn−0.63Er alloy after solution treatment at different temperatures for 10 h: (a) 440 °C; (b) 460 °C; (c) 480 °C; (d) 500 °C

Figure 6 shows the TEM images of the W-phase and the corresponding SAED pattern found in the alloy solid after solution treatment at 480 °C for 10 h. The W-phase with an elliptical morphology precipitates at the matrix nonuniformly. The size of the W-phase particles is about 140 nm in length and 80 nm in width. It has been reported that the W-phase is more stable than the I-phase. Therefore, the W-phase is inclined to form during the solid solution treatment in order to reduce the energy of the system [25]. However, the nano-scale W-phase is first reported in the Mg−Zn−Er series alloys. The generative mechanism of the W-phase precipitated during the solid solution treatment will be researched in the further investigation.

3.3 Aging responses of solution-treated alloys and microstructures of aging alloys

Figure 7 shows the aging behaviors of the solid solution-treated alloys at 175 °C for 6−100 h. The results show that the peak hardness increases as the solution-treated temperature increases in the range of 440−480 °C. However, the peak hardness of the alloy after solution

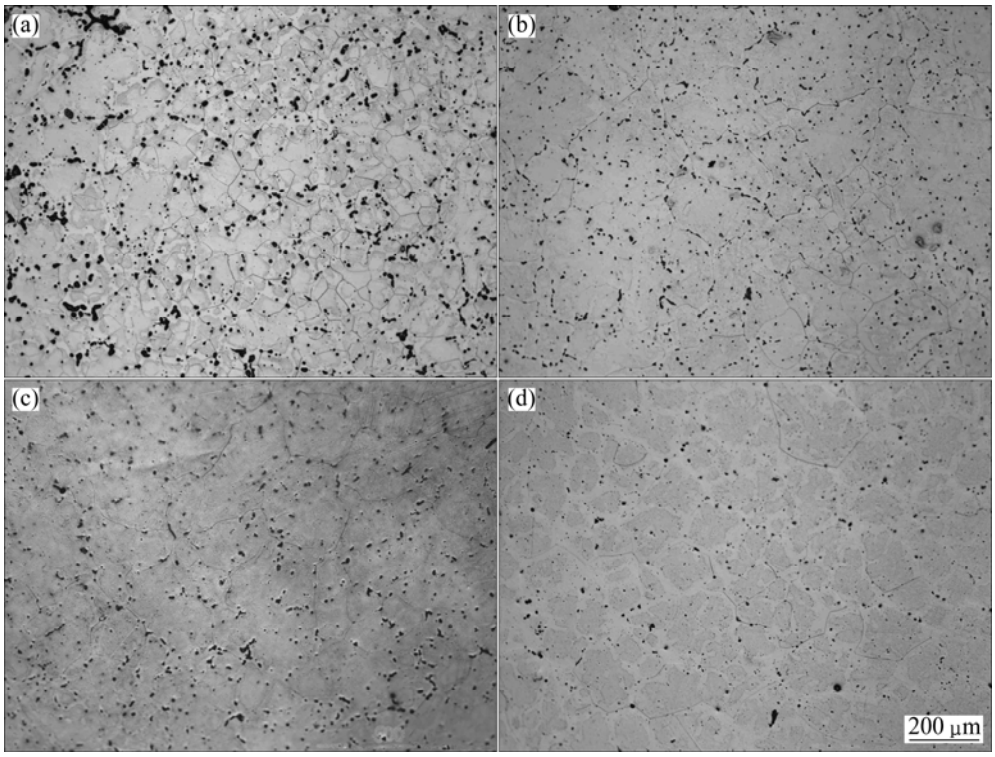

Fig. 5 OM images of Mg−5Zn−0.63Er alloy after solution treatment at different temperatures for 10 h: (a) 440 °C; (b) 460 °C; (c) 480 °C; (d) 500 °C

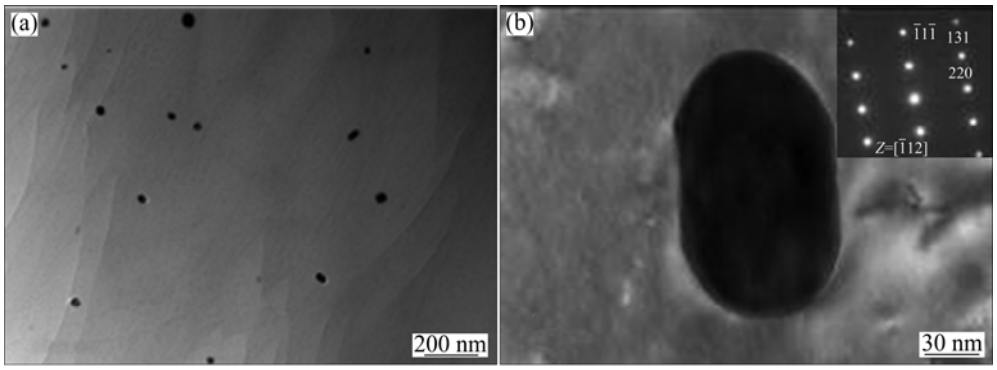

Fig. 6 TEM images of W-phase in Mg−5Zn−0.63Er alloy after solid solution treatment at 480 °C for 10 h: (a) Dispersive W-phases; (b) Bright field micrograph for one example of W-phase precipitate

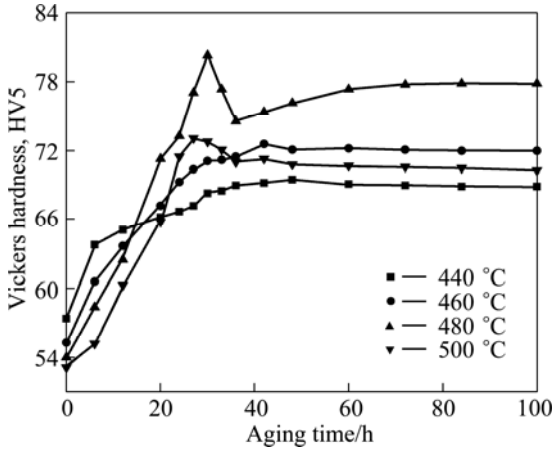

Fig. 7 Aging behaviors of solution-treated alloys aged at 175 °C for 6−100 h

treatment at 500 °C decreases. Meanwhile, the time arriving at peak hardness gets shortened as the solution temperature increases. The peak hardness values of the specimens solution-treated at 440, 460, 480 and 500 °C are obtained after 48, 42, 30 and 27 h, respectively, and the corresponding peak hardness values are 69.5, 72.6, 80.3 and 73.1, respectively. The alloy solution-treated at 480 °C for 10 h exhibits the highest peak hardness.

Figure 8 shows the OM image of the peak-aged Mg−5Zn−0.63Er alloy which was solid solution-treated at 480 °C and aged at 175 °C for 30 h. It is shown that the microstructures are constituted by the equiaxed grains. Large numbers of tiny particles are found at the grain boundaries and the matrix. In order to investigate the strengthening phase precipitated during the aging

process, the TEM observation was conducted on the peak-aged alloy (solid solution-treated at 480 °C), as shown in Fig. 9. It is indicated that, large amounts of rod-like $MgZn_2$ phases precipitate together with lots of disc-like $MgZn_2$ at the matrix. As previously reported, the rod-like $MgZn_2$ phase in the ZK60−Y(6.3% Zn, 2.0% Zr and 1% Y) alloy would form during T6 heat treatment [26]. The habit plane of the $MgZn_2$ precipitates is $(\bar{1}20)$ plane, with its long axis parallel to the $[0001]_{Mg}$ [27,28]. The $MgZn_2$ precipitates are responsible for the enhancement of the peak hardness.

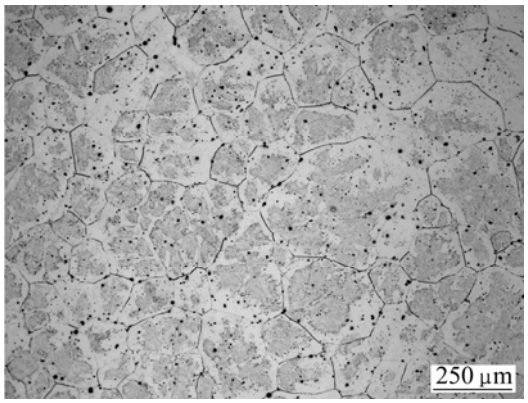

Fig. 8 OM image of Mg−5Zn−0.63Er alloy solid solution-treated at 480 °C and aged at 175 °C for 30 h

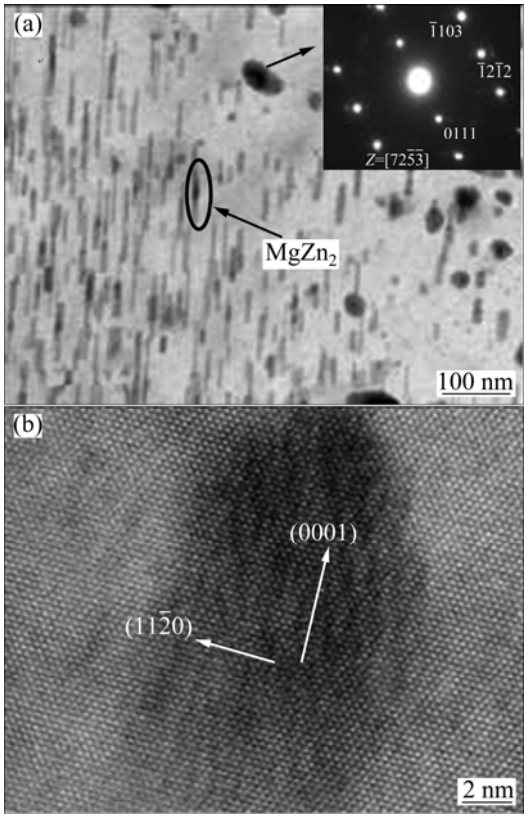

Fig. 9 TEM images of peak-aged alloy after solution treatment at 480 °C and aging at 175 °C for 30 h: (a) Bright field image; (b) High resolution TEM image of $MgZn_2$ in (a)

3.4 Mechanical properties of Mg−5Zn−0.63Er alloys

Figure 10 shows the tensile properties of the Mg−5Zn−0.63Er alloys. It is shown that the ultimate tensile strength (UTS) and tensile yield strength (TYS) of the cast alloy are approximately 223 MPa and 112.5 MPa, respectively. The solution treatment at 480 °C for 10 h leads to a small reduction of the tensile strength. The UTS and TYS of the solution-treated alloy are about 206.8 MPa and 106 MPa, respectively, while the elongation increases up to 13.6%. However, the tensile strength of the alloy after T6 treatment ((480 °C, 10 h)+ (175 °C, 30 h)) is greatly improved. The UTS and TYS are 261 MPa and 124 MPa, respectively, companying with an elongation of 10.5%.

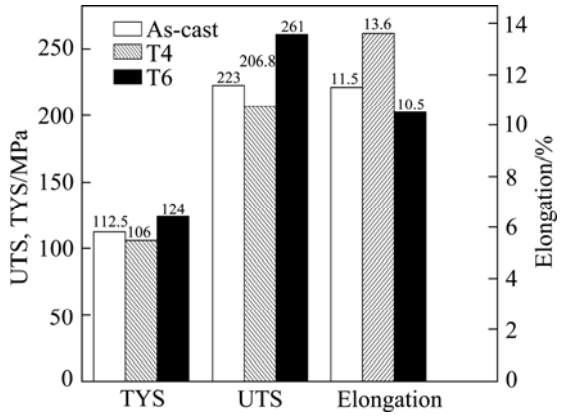

Fig. 10 Mechanical properties of Mg−5Zn−0.63Er alloys under different conditions at room temperature

As suggested above, the as-cast Mg−5Zn−0.63Er alloy displays a better elongation of 11.5%, compared with other as-cast alloys, such as Mg−RE, AZ series alloys [25,29]. It is thought that the excellent plastic deformation is mainly related with the existence of the I-phase [30]. The quasiperiodic lattice of the I-phase provides a stable interface with the matrix, and its high symmetry offers greater chances that one of its faces will have a local atomic match with one of the planes of the matrix [30,31]. In addition, the I-phase distributing at the grain boundaries creates a banded structure, which would block the movement of the dislocations, prevent the migration of the grain boundaries and inhibit the grain growth. However, the melting point of the I-phase is about 450 °C [32], therefore, the high solution temperature will make the I-phase dissolve into the matrix. It is obviously shown that the volume fraction of the secondary phases decreases as the solution temperature increases. Besides, the average grain size of the alloy becomes coarse at the high temperature. According to the well-known Hall-Petch relationship, strengthening of the alloy depends on the grain size [33]. Meanwhile, the precipitates formed during the heat treatment also have an important effect on improving

tensile strength [34]. In conclusion, the TYS of the solution-treated Mg−5Zn−0.63Er alloy decreases to 106 MPa due to the grain growth and the disappearance of the secondary phases mainly including the I-phase and the Mg_4Zn_7 phase. The TEM observation of the peak-aged specimen suggests that the presence of the rod-like $MgZn_2$ phase is responsible for the enhancement of the mechanical properties, and the phase mostly distributing at the matrix uniformly and densely plays an important role in strengthening the alloys. As reported previously, the $MgZn_2$ precipitates can act as semi-fibers so that the interface between $MgZn_2$ precipitates and the matrix can hold more loads when the basal of grains is parallel to the tensile direction, which can increase the mechanical properties. In the investigation, the T6 treatment advances the UTS of the Mg−5Zn−0.63Er alloy greatly without consumption of the elongation, and the value of the UTS is enhanced by 38 MPa. The addition of rare earth elements has an effect on improving aging behaviors of the Mg−Zn alloys, but the improvement is unobvious. However, the present investigation suggests that the trace addition of 0.63% Er is beneficial to the tensile strength and aging behaviors. The improvement of the tensile strength and aging response is great, compared with that of the other rare earth elements. The UTS of the alloy after peak aging treatment is high, and its value is about 261 MPa.

4 Conclusions

1) The as-cast Mg−5Zn−0.63Er alloy mainly consists of primary α-Mg solid solution, $Mg_3Zn_6Er_1$ phase (I-phase) and little fine granular-shaped Mg_4Zn_7 phase.

2) The I-phase dissolves during the solution treatment, and disappears completely after solution treatment at 480 °C for 10 h. The alloy solution-treated at 480 °C for 10 h displays a better aging behavior than the alloy solution treated at other temperatures. As a result, the proper heat treatment condition is found as (480 °C, 10 h)+(175 °C, 30 h).

3) The Mg−5Zn−0.63Er alloy after T6 treatment ((480 °C, 10 h)+(175 °C, 30 h)) exhibits higher tensile strength due to the presence of rod-like $MgZn_2$ phase. The values of UTS and TYS are 261 MPa and 124 MPa, respectively.

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热处理对 Mg−5Zn−0.63Er 合金显微组织及力学性能的影响

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摘 要：通过不同的热处理工艺研究含有准晶 I 相的铸态 Mg−5Zn−0.63Er(质量分数，%)合金的显微组织演变。结果表明：合金在 480 °C 固溶 10 h 后，除有 W 相颗粒析出外，准晶 I 相几乎全部固溶在基体中。固溶态 Mg−5Zn−0.63Er 合金在 175 °C 下时效 6~10 h。合金在峰时效态的抗拉强度约为 261 MPa、伸长率为 10.5%。合金拉伸强度的提高归因于杆状 $MgZn_2$ 相的析出。

关键词：Mg−5Zn−0.63Er 合金；稀土；铒；热处理；显微组织；力学性能

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